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Crystal structure of 2-ethylquinazoline-4(3*H*)-thione

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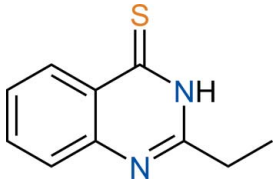
In the title compound, $C_{10}H_{10}N_2S$, all non-H atoms are almost coplanar [maximum deviation = 0.103 (1) Å]. In the crystal, N—H···S interactions form $R_2^2(8)$ rings linking pairs of molecules related by inversion. The molecular pairs are stacked along [100]. A herringbone arrangement of pairs in the [010] direction forms layers parallel to (010).

Keywords: crystal structure; N—H···S interactions; quinazoline-4(3*H*)-thione; hydrogen-bonded dimers; herringbone arrangement.

CCDC reference: 1014729

1. Related literature

For the synthesis of quinazoline-4(3*H*)-thiones, see: Bogert *et al.* (1903); Zoltewicz & Sharpless (1976); Segarra *et al.* (1998); El-Hiti (2004); Ozturk *et al.* (2007); El-Hiti *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_{10}H_{10}N_2S$ M_r = 190.26

Orthorhombic, *Pbca*
a = 5.8231 (3) Å
b = 14.3214 (6) Å
c = 21.7365 (8) Å
V = 1812.71 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 150 K
0.41 × 0.24 × 0.15 mm

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.780, T_{max} = 1.000
7795 measured reflections
2240 independent reflections
1973 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.087
S = 1.03
2240 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.88	2.53	3.3854 (11)	166

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: XU5804).

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supporting information

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S1. Structural commentary

The 2-ethyl-3*H*-quinazoline-4-thione molecule (Fig 1) is almost planer (apart from the ethyl hydrogens) with the ethyl group being twisted from the quinazoline-4-thione plane by 8.7 (2)°. N—H···S interactions form $R_2^2(8)$ rings to link pairs of molecules related by inversion. The pairs of molecules are stacked parallel to the *a*-axis (Fig 2). Adjacent pairs pack in a herring bone arrangement in the [010] direction to form layers parallel to the (010) plane. 2-(Substituted alkyl)-3*H*-quinazoline-4-thione derivatives can be obtained from double lithiation of 2-alkyl-3*H*-quinazoline-4-thiones followed by reactions with electrophiles, including alkyl iodides, at low temperature in anhydrous THF (El-Hiti, 2004). Also, 3*H*-quinazoline-4-thiones are produced from the corresponding 3*H*-quinazoline-4-ones using phosphorus pentasulfide (Bogert *et al.*, 1903; Ozturk *et al.*, 2007; El-Hiti *et al.*, 2011) or Lawesson's reagent (Segarra *et al.*, 1998). 3*H*-Quinazoline-4-thiones have also been synthesized in one-step from reaction of 2-aminobenzonitriles and thioamides in the presence of hydrogen bromide in various solvents on a steam bath for 1–4 h (Zoltewicz & Sharpless, 1976).

S2. Synthesis and crystallization

2-Ethyl-3*H*-quinazoline-4-thione was obtained in 92% yield from double lithiation of 2-methyl-3*H*-quinazoline-4-thione with *n*-butyllithium at 78 °C in anhydrous THF under nitrogen followed by reaction with iodomethane (El-Hiti, 2004). Crystallization from methanol gave the title compound as yellow crystals. The NMR and low and high resolution mass spectra for the title compound were consistent with those reported (El-Hiti, 2004).

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were placed in calculated positions with C—H = 0.95 and 0.98Å and refined in riding mode, U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2Ueq(C) for aromatic H atoms

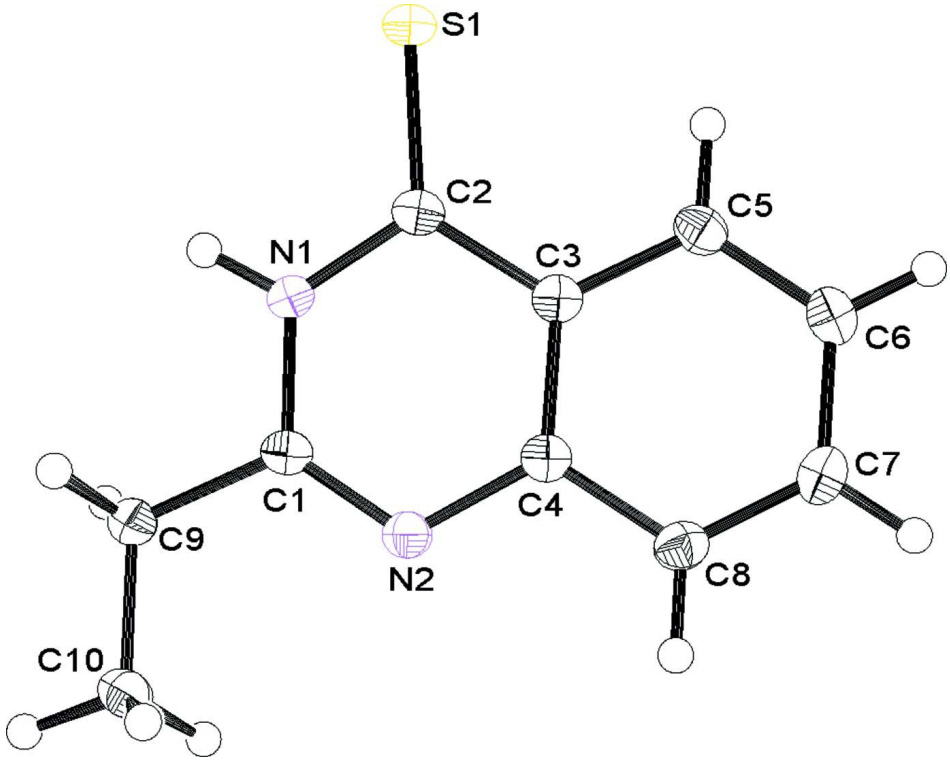

Figure 1

A molecule of the title compound showing atom labels and 50% probability displacement ellipsoids for non-H atoms.

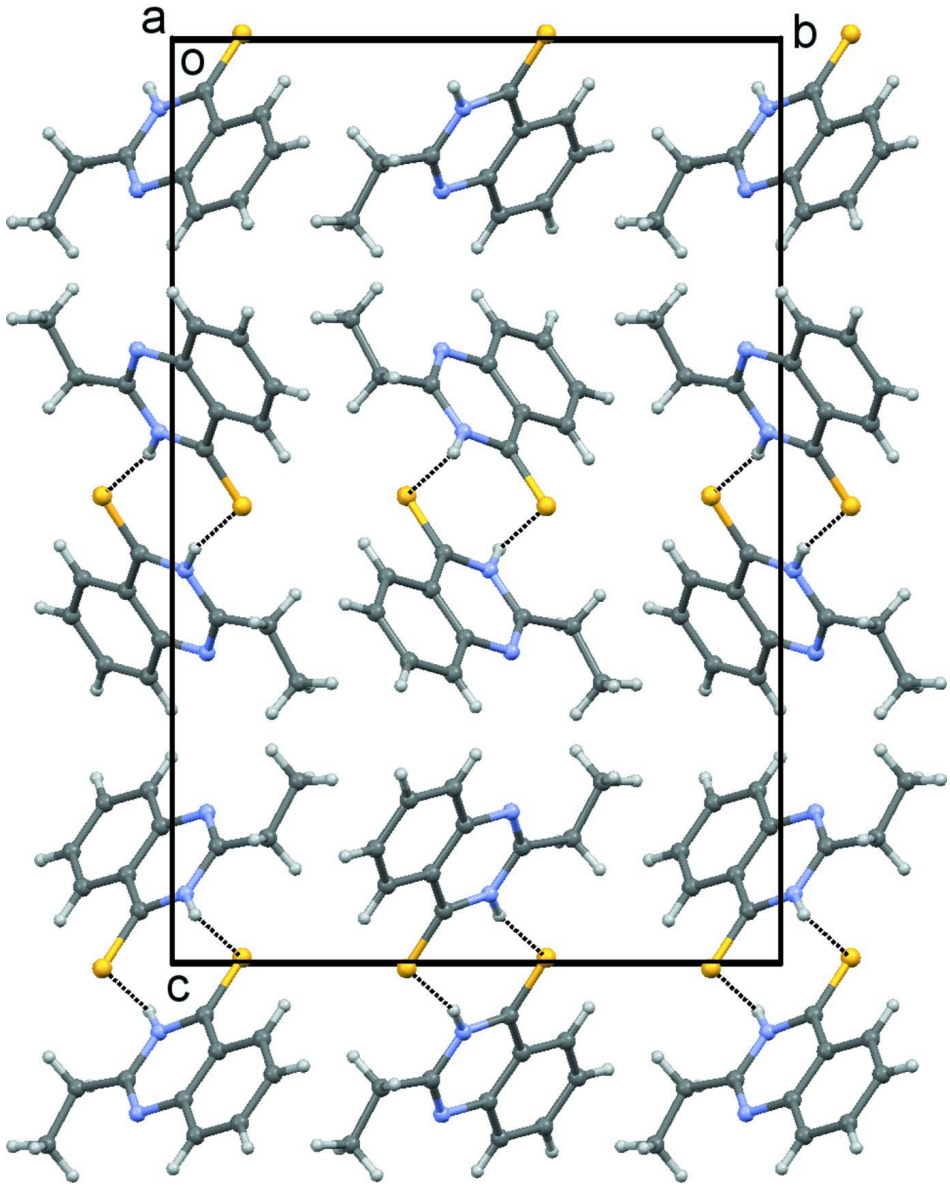

Figure 2

Crystal structure packing showing N—H···S contacts as dotted lines.

2-Ethylquinazoline-4(3*H*)-thione

Crystal data

$C_{10}H_{10}N_2S$
$M_r = 190.26$
Orthorhombic, *Pbca*
$a = 5.8231\ (3)$ Å
$b = 14.3214\ (6)$ Å
$c = 21.7365\ (8)$ Å
$V = 1812.71\ (14)$ Å^3
$Z = 8$
$F(000) = 800$
$D_x = 1.394$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3617 reflections
$\theta = 3.9$–$29.3°$
$\mu = 0.31$ mm^{-1}
$T = 150$ K
Plate, yellow
0.41 × 0.24 × 0.15 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.780, T_{max} = 1.000
7795 measured reflections
2240 independent reflections
1973 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 29.8°, θ_{min} = 3.0°
h = −6→7
k = −19→14
l = −23→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.087
S = 1.03
2240 reflections
119 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.7981P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.7433 (2)	0.57478 (9)	0.62537 (6)	0.0185 (3)
C2	0.6649 (2)	0.44643 (8)	0.55604 (5)	0.0178 (2)
C3	0.4705 (2)	0.42557 (8)	0.59550 (5)	0.0177 (3)
C4	0.4368 (2)	0.48131 (8)	0.64832 (5)	0.0183 (3)
C5	0.3171 (2)	0.35262 (9)	0.58239 (6)	0.0209 (3)
H5	0.3379	0.3158	0.5465	0.025*
C6	0.1366 (2)	0.33425 (9)	0.62146 (6)	0.0236 (3)
H6	0.0337	0.2845	0.6127	0.028*
C7	0.1046 (2)	0.38917 (9)	0.67440 (6)	0.0240 (3)
H7	−0.0197	0.3760	0.7013	0.029*
C8	0.2513 (2)	0.46175 (9)	0.68761 (6)	0.0218 (3)
H8	0.2272	0.4987	0.7233	0.026*
C9	0.8998 (2)	0.65696 (9)	0.63452 (6)	0.0227 (3)
H9A	0.8734	0.7023	0.6009	0.027*
H9B	1.0609	0.6354	0.6316	0.027*
C10	0.8668 (3)	0.70632 (9)	0.69567 (6)	0.0246 (3)
H10A	0.7058	0.7252	0.7000	0.037*
H10B	0.9652	0.7618	0.6972	0.037*
H10C	0.9080	0.6639	0.7293	0.037*
N1	0.78973 (18)	0.52156 (7)	0.57402 (5)	0.0188 (2)
H1	0.9086	0.5374	0.5513	0.023*

N2	0.57662 (19)	0.55697 (7)	0.66273 (5)	0.0199 (2)
S1	0.73978 (6)	0.38502 (2)	0.49367 (2)	0.02214 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0210 (6)	0.0177 (6)	0.0169 (6)	0.0022 (5)	−0.0006 (4)	−0.0010 (5)
C2	0.0203 (6)	0.0164 (5)	0.0166 (5)	0.0039 (5)	−0.0026 (5)	0.0008 (4)
C3	0.0194 (6)	0.0171 (6)	0.0167 (5)	0.0027 (5)	−0.0019 (5)	0.0020 (4)
C4	0.0197 (6)	0.0178 (6)	0.0172 (5)	0.0019 (5)	−0.0012 (4)	0.0010 (5)
C5	0.0237 (7)	0.0189 (6)	0.0202 (6)	0.0008 (5)	−0.0032 (5)	−0.0006 (5)
C6	0.0238 (7)	0.0212 (6)	0.0258 (6)	−0.0033 (5)	−0.0029 (5)	0.0012 (5)
C7	0.0213 (7)	0.0264 (7)	0.0244 (6)	−0.0013 (5)	0.0028 (5)	0.0038 (5)
C8	0.0242 (7)	0.0226 (6)	0.0186 (6)	0.0015 (5)	0.0016 (5)	0.0002 (5)
C9	0.0238 (7)	0.0208 (6)	0.0233 (6)	−0.0033 (5)	0.0041 (5)	−0.0040 (5)
C10	0.0292 (7)	0.0233 (6)	0.0214 (6)	−0.0047 (5)	0.0001 (5)	−0.0040 (5)
N1	0.0188 (5)	0.0194 (5)	0.0182 (5)	−0.0003 (4)	0.0030 (4)	−0.0024 (4)
N2	0.0220 (6)	0.0191 (5)	0.0187 (5)	−0.0006 (4)	0.0015 (4)	−0.0012 (4)
S1	0.0244 (2)	0.02191 (18)	0.02011 (17)	0.00029 (12)	0.00293 (12)	−0.00599 (12)

Geometric parameters (Å, º)

C1—N2	1.2908 (16)	C6—C7	1.4061 (19)
C1—N1	1.3784 (16)	C6—H6	0.9500
C1—C9	1.5017 (18)	C7—C8	1.3757 (19)
C2—N1	1.3560 (16)	C7—H7	0.9500
C2—C3	1.4514 (17)	C8—H8	0.9500
C2—S1	1.6737 (12)	C9—C10	1.5177 (17)
C3—C5	1.4038 (18)	C9—H9A	0.9900
C3—C4	1.4119 (16)	C9—H9B	0.9900
C4—N2	1.3910 (16)	C10—H10A	0.9800
C4—C8	1.4054 (18)	C10—H10B	0.9800
C5—C6	1.3766 (19)	C10—H10C	0.9800
C5—H5	0.9500	N1—H1	0.8800
N2—C1—N1	123.21 (12)	C6—C7—H7	119.6
N2—C1—C9	121.86 (11)	C7—C8—C4	120.08 (12)
N1—C1—C9	114.92 (11)	C7—C8—H8	120.0
N1—C2—C3	114.27 (11)	C4—C8—H8	120.0
N1—C2—S1	120.71 (10)	C1—C9—C10	113.84 (11)
C3—C2—S1	125.01 (10)	C1—C9—H9A	108.8
C5—C3—C4	119.83 (12)	C10—C9—H9A	108.8
C5—C3—C2	121.97 (11)	C1—C9—H9B	108.8
C4—C3—C2	118.19 (11)	C10—C9—H9B	108.8
N2—C4—C8	117.93 (11)	H9A—C9—H9B	107.7
N2—C4—C3	122.83 (11)	C9—C10—H10A	109.5
C8—C4—C3	119.22 (12)	C9—C10—H10B	109.5
C6—C5—C3	120.19 (12)	H10A—C10—H10B	109.5

C6—C5—H5	119.9	C9—C10—H10C	109.5
C3—C5—H5	119.9	H10A—C10—H10C	109.5
C5—C6—C7	119.94 (12)	H10B—C10—H10C	109.5
C5—C6—H6	120.0	C2—N1—C1	124.53 (11)
C7—C6—H6	120.0	C2—N1—H1	117.7
C8—C7—C6	120.73 (13)	C1—N1—H1	117.7
C8—C7—H7	119.6	C1—N2—C4	116.89 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.88	2.53	3.3854 (11)	166

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.